

# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 733107	<b>FOR FURTHER ACTION</b>		See Form PCT/IPEA/416
International application No. <b>PCT/AU2004/001608</b>	International filing date ( <i>day/month/year</i> ) 19 November 2004	Priority date ( <i>day/month/year</i> ) 19 November 2003	
International Patent Classification (IPC) or national classification and IPC  Int. Cl.  <div style="display: flex; justify-content: space-between;"> <div style="width: 30%;"> <b>C04B 7/46</b> (2006.01)  <b>B28B 21/02</b> (2006.01)  <b>C01B 33/24</b> (2006.01)         </div> <div style="width: 30%;"> <b>C01B 33/32</b> (2006.01)  <b>C04B 22/06</b> (2006.01)  <b>C04B 28/00</b> (2006.01)         </div> <div style="width: 30%;"> <b>C04B 28/02</b> (2006.01)  <b>C04B 28/06</b> (2006.01)         </div> </div>			
Applicant <b>ROCLA PTY LTD et al</b>			

1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 3 sheets, including this cover sheet.
3. This report is also accompanied by ANNEXES, comprising:
  - a. ☒ (*sent to the applicant and to the International Bureau*) a total of 5 sheets, as follows:
 

☐ sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).  
☐ sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.
  - b. ☐ (*sent to the International Bureau only*) a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or table related thereto, in electronic form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).
4. This report contains indications relating to the following items:
 

☒ Box No. I Basis of the report  
☐ Box No. II Priority  
☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability  
☐ Box No. IV Lack of unity of invention  
☒ Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement  
☐ Box No. VI Certain documents cited  
☐ Box No. VII Certain defects in the international application  
☐ Box No. VIII Certain observations on the international application

Date of submission of the demand 19 September 2005	Date of completion of this report 13 December 2005
Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustalia.gov.au Facsimile No. (02) 6285 3929	Authorized Officer  <b>WARREN TAYLOR</b> Telephone No. (02) 6283 2229

**Box No. I Basis of the report**

1. With regard to the language, this report is based on:
- ☒ The international application in the language in which it was filed
- ☐ A translation of the international application into \_\_\_\_\_, which is the language of a
- translation furnished for the purposes of:
- ☐ international search (under Rules 12.3(a) and 23.1 (b))
- ☐ publication of the international application (under Rule 12.4(a))
- ☐ international preliminary examination (Rules 55.2(a) and/or 55.3(a))
2. With regard to the elements of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report)*:
- ☐ the international application as originally filed/furnished
- ☒ the description:
- pages **1, 5-17** as originally filed/furnished
- pages\* **2-3** received by this Authority on **10 November 2005** with the letter of **10 November 2005**
- pages\* **4** received by this Authority on **19 September 2005** with the letter of **19 September 2005**
- ☒ the claims:
- pages **19 & 21** as originally filed/furnished
- pages\* as amended (together with any statement) under Article 19
- pages\* **18 & 20** received by this Authority on **10 November 2005** with letter of **10 November 2005**
- pages\* received by this Authority on with the letter of
- ☐ the drawings:
- pages as originally filed/furnished
- pages\* received by this Authority on with the letter of
- pages\* received by this Authority on with the letter of
- ☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing.
3. ☐ The amendments have resulted in the cancellation of:
- ☐ the description, pages
- ☐ the claims, Nos.
- ☐ the drawings, sheets/figs
- ☐ the sequence listing (*specify*):
- ☐ any table(s) related to the sequence listing (*specify*):
4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).
- ☐ the description, pages
- ☐ the claims, Nos.
- ☐ the drawings, sheets/figs
- ☐ the sequence listing (*specify*):
- ☐ any table(s) related to the sequence listing (*specify*):

\* If item 4 applies, some or all of those sheets may be marked "superseded."

**Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Claims 1-27	YES
	Claims	NO
Inventive step (IS)	Claims 1-27	YES
	Claims	NO
Industrial applicability (IA)	Claims 1-27	YES
	Claims	NO

**2. Citations and explanations (Rule 70.7)**

The current application is directed to geopolymer based concrete and a method of casting said concrete to form products such as pipes, poles, railway sleepers and the like.

The problem to solve appears to reside in providing a 'no slump' geopolymer concrete having a better plastic consistency of freshly prepared geopolymer-based concrete in order to increase the working time of said concrete to produce pipes, poles and the like.

In light of the comments made in Box VIII of this opinion it is considered the following documents are most relevant to the current application;

D1 - WO 2003/078349 (previously D3)

D1 appears to be the closest art in regard to the amended application.

This document discloses each of the features contained in proposed claim 1 and particularly a  $\text{SiO}_2:\text{M}_2\text{O}$  ratio that overlaps the ratio now prescribed in claim 1. Said geopolymer concrete composition can be formed and cast into a mould whereby consolidation of said geopolymer concrete occurs in the mould.

The applicant asserts the water content of D1 is such that an unstable concrete is formed (due to a high slump -- in excess of 200mm) and therefore different to the current application invention. On this basis the currently proposed application appears to be novel over D1.

**NOVELTY (N) Claims 1-27**

Proposed independent claim 1 defines combining an aluminosilicate component, an alkali or alkaline metal earth silicate component, an alkali or alkaline earth metal hydroxide, aggregate and water - the composition having a  $\text{SiO}_2:\text{M}_2\text{O}$  ratio of between 0.8-1.2 and where M is an alkali metal. The composition is then cast into any mould, where said composition is consolidated in the mould.

It is considered the disclosure of D1 does not deprive the currently proposed application claims of novelty.

**INVENTIVE STEP (IS) Claims 1-27**

Accordingly, since the disclosure of D1 does not deprive the proposed application claims of novelty, this document also does not deprive the proposed claims of an inventive step. It is also considered the skilled artisan would not find the disclosure of D1 useful in facilitating the solution to the current application problem.

said to provide rapid strength by use of a  $\text{SiO}_2:\text{Na}_2\text{O}$  ratio of about 0.20:1 to about 0.75:1 (preferably about 0.5:1 to about 0.6:1).

5 [0006] Hardjito et al of Curtin University of Technology studied the effect of different mix design variables in their paper "The Engineering Properties of Geopolymer Concrete" (Concrete in Australia, Dec 2002 – Feb 2003, pp24-29). The geopolymer concrete is prepared by the method of mixing the aggregates and fly ash and adding the alkaline solution to this dry mix. Hardjito et al report that the compressive strength of geopolymer concrete, unlike OPC concrete does not increase with aging.  
10 In their subsequent work Hardjito et al study the use of naphthalene based superplasticizer to delay the onset of curing and allow the concrete to be handled for up to 120 minutes without any sign of setting.

[0007] The present inventor found that geopolymer concrete, of previously reported  
15 composition and prepared by previously reported techniques, cannot be used with the usual manufacturing processes for Portland Cement concrete pipes, poles and the like because the working time is too short. These manufacturing techniques for Portland Cement concrete require the use of concrete with a 'No Slump' consistency and the inventor found that a low fluid content in geopolymer concrete leads to a  
20 shortened working time. A further shortening of the working time was caused by the vibration and compaction techniques used in the manufacturing process and these two factors made it impossible to form geopolymer products of acceptable appearance and with properties that allow them to pass the standard requirements. This had not been expected as the work life of Ordinary Portland Cement products is  
25 not accelerated in this way.

[0008] Geopolymer concrete needs to be cured at elevated temperatures to accelerate the hardening reactions and we found that products of acceptable quality could only be produced if the plastic consistency of the fresh concrete was  
30 maintained during the forming and transport of the products to the curing chambers. Transport of the products after they had lost this plastic consistency can result in cracking and a reduction in the final strength of the product. If the manufacture of these products is to be performed on a continuous basis then it is also necessary to maintain the plastic consistency for the time required to make at

also necessary to maintain the plastic consistency for the time required to make at least two successive batches of concrete.

5 [0009] In many of the casting techniques previously used for Ordinary Portland Cement based concrete the concrete is cast in a relatively dry form. Such concrete is often referred to as "no-slump" concrete as the concrete does not exhibit any measurable slump when placed on a hard flat surface. No-slump concrete based on ordinary Portland cement is used in centrifugal casting of pipes and other dry compaction casting methods. As a consequence of the rapid setting of geopolymer concrete when it is subject to such consolidation methods we found that casting of products presented considerable practical problems. It made it extremely difficult to transfer the laboratory scale observations reported in the literature to commercial scale manufacture of products as the literature does not recognise or allow for the change in the properties of geopolymer concrete which are brought about by subjecting the geopolymer to the conventional consolidation techniques used in manufacture of pipe and the like products.

20 The discussion of documents, acts, materials, devices, articles and the like is included in this specification solely for the purpose of providing a context for the present invention. It is not suggested or represented that any or all of these matters formed part of the prior art base or were common general knowledge in the field relevant to the present invention as it existed before the priority date of each claim of this application.

### Summary

25 [0010] We have now found that geopolymer concrete may be used in preparing pipe and other consolidated moulded products by using a geopolymer concrete which has a "no-slump" consistency and a metal silicate and metal hydroxide component which together provide a weight ratio of  $\text{SiO}_2/\text{M}_2\text{O}$  of at least 0.8 where M is an alkali metal or alkaline earth metal ( $1/2 \text{ M}$ ) and preferably is an alkali metal such as sodium or potassium.

30 [0011] A method of forming a geopolymer moulded product comprising: forming a geopolymer concrete composition comprising a mixture of an alumino silicate component an alkali or alkaline earth metal silicate component, an alkali or alkaline earth metal hydroxide, aggregate and water wherein the water content is insufficient to provide a slumped concrete and the ratio of  $\text{SiO}_2$  to  $\text{M}_2\text{O}$  wherein M is an alkali metal is in the range of from 0.8 to 1.2; casting the concrete into a mould; and  
35 subjecting the moulded concrete to consolidation in the mould.

[0012] We also found that concrete with acceptable working time could be obtained by restricting the water added at the start of the mixing sequence. It is usual practise to begin a mixing cycle by adding the aggregate components to the mixer and those aggregates will typically be added in an 'as received' moisture condition. When this usual practise is followed with a geopolymer concrete mix, the water contributed by the aggregate was found to shorten the working time. To overcome this problem we prefer to precondition the aggregate in a way that will restrict the water addition at the start of the mixing cycle.

[0013] We also found that more uniform workability could be obtained, that would allow the concrete to be compacted more easily and produce a better surface finish by using a certain order of addition for the components. The method of preparation comprised forming a mixture of at least part of the aggregate with a metal hydroxide and combining this mixture with an aluminosilicate binder followed by a metal silicate and a final water addition.

[0014] The composition and process of the invention is particularly suited to use in the preparation of pipe.

#### Detailed Description

[0015] We found that by manipulating these aspects of the invention that adequate working time could be achieved, which would allow geopolymer concrete to be used for making pipes, poles and the like by the normal manufacturing techniques. The manipulation of these aspects still allows the concrete to achieve rapid strength growth during the curing process and produce products of typical dimensions that comply with the appropriate standard requirements.

[0016] Concrete used for the production of pipes, poles and the like has a very stiff consistency and it is generally referred to as being 'No Slump' concrete. No slump

## Claims

1. A method of forming a geopolymer moulded product comprising: forming a geopolymer concrete composition comprising a mixture of an alumino silicate component an alkali or alkaline earth metal silicate component, an alkali or alkaline earth metal hydroxide, aggregate and water wherein the water content is insufficient to provide a slumped concrete and the weight ratio of  $\text{SiO}_2$  to  $\text{M}_2\text{O}$  wherein M is an alkali metal is in the range of from 0.8 to 1.2; and casting the concrete into a mould; and subjecting the moulded concrete to consolidation in the mould.
2. A method according to claim 1 wherein a metal M is at least one of sodium and potassium.
3. A method according to claim 1 wherein the ratio of  $\text{SiO}_2$  to  $\text{M}_2\text{O}$  is at least 0.9.
4. A method according to claim 1 wherein the ratio of  $\text{SiO}_2$  to  $\text{M}_2\text{O}$  is at least 0.95.
5. A method according to claim 1 wherein  $\text{M}_2\text{O}$  is  $\text{Na}_2\text{O}$  and the ratio of  $\text{SiO}_2/\text{Na}_2\text{O}$  is in the range of 0.9 to 1.2.
6. A method according to claim 1 wherein at 15 minutes after mixing the concrete has a Vebe time in the range of from 15 to 40 seconds.
7. A method according to claim 6 wherein at 30 minutes the concrete has a Vebe time in the range of 15 to 50 seconds and at 45 minutes the concrete has a Vebe time of from 15 to 60 seconds.
8. A method according to claim 1 used in the moulding of concrete products.
9. A method according to claim 1 used in the formation of moulded pipe by methods selected from the group consisting of centrifugal processes, roller suspension process and vertical casting processes.

component with the metal hydroxide and combining the mixture of metal hydroxide and at least part of the aggregate with a binder comprising aluminosilicate material and an activator comprising metal silicate.

- 5 17. A method of preparing a geopolymer concrete according to claim 16 wherein at least 50% of the total aggregate component is present in the mixture with the aggregate and metal hydroxide.
- 10 18. A method of preparing a geopolymer concrete according to claim 16 wherein the aggregate mixed with the metal hydroxide has a water content of less than 0.8 % of the total mass of components.
- 15 19. A method of preparing a geopolymer concrete according to claim 16 wherein the geopolymer concrete composition is cast into a mould and compacted into the mould.
- 20 20. A method according to claim 16 wherein the concrete composition is cast into a pipe mould by a process selected from the group consisting of centrifugal pipe process, roller suspension process and vertical casting process.
- 25 21. A method according to claim 16 wherein the concrete is cast into a pipe mould by a process selected from centrifugal process and roller suspension process.
- 30 22. A method according to claim 16 wherein the geopolymer concrete is a no slump concrete.
23. A method according to claim 16 wherein the ratio of sand to stone in the composition is in the range of from 1:1.5 to 1:2.
24. A method according to claim 16 wherein water is present in the mixture of at least part of the aggregate component and metal hydroxide and further water is added with the remaining components and wherein the